HERCULES

# Klucel®
# hydroxypropylcellulose

Physical and Chemical Properties

Aqualon

**HERCULES INCORPORATED**
Aqualon Division
Hercules Plaza
1313 North Market Street
Wilmington, DE 19894-0001
(302) 594-5000
www.aqualon.com

ORDER PLACEMENT INFORMATION
(800) 334-8426

PRODUCT AND TECHNICAL INFORMATION
(800) 345-0447

Aqualon

# KLUCEL® HYDROXYPROPYLCELLULOSE

CONTENTS PAGE

# KLUCEL® HYDROXYPROPYLCELLULOSE

KLUCEL® hydroxypropylcellulose (HPC) is a nonionic water-soluble cellulose ether with a remarkable combination of properties. It combines organic solvent solubility, thermoplasticity, and surface activity with the aqueous thickening and stabilizing properties characteristic of other water-soluble cellulose polymers available from Hercules Incorporated, Aqualon Division. KLUCEL films are flexible without plasticizers and non-tacky at high humidity.

The information in this booklet presents the physical and chemical properties of KLUCEL as developed in our research and plant facilities. Also included is information about the behavior of KLUCEL with many of the materials that would be used in most applications. A guide to regulatory status and toxicological studies is provided for convenient reference. The Appendix gives information about Aqualon test methods for viscosity, moisture, and ash content.

To help the reader identify the versatile uses for this water-soluble polymer, a representative listing has been developed and is presented on the following page. Many of these uses for KLUCEL are discussed in detail in technical literature available from your Aqualon sales representative by request, and/or from our website at www.Aqualon.com.

# APPLICATIONS

Applications for KLUCEL® Hydroxypropylcellulose

| Types of Uses | Specific Applications | Properties Utilized |
|---|---|---|
| Adhesives | Solvent-based<br>Hot-melt | Thickener<br>Thermoplastic |
| Aerosols | Food – whipped toppings<br>Emulsions – cosmetics<br>Solvent-based | Stabilizer, foaming aid<br><br>Film-former, binder |
| Binder | Burnout types<br>Electrical insulators<br>Ceramic glazes<br>Alcohol core-wash compounds<br>Matrix board manufacture | Ready burnout, low residue,<br>solvent-soluble<br><br>Thickener, binder suspending agent<br>Solvent-soluble |
| Coatings | Edible food coatings<br>Textile and paper coatings<br>Film coatings | Glaze, oil- and oxygen-barrier<br>Solvent-soluble film-former, oil- and<br>fat-barrier, heat-sealable |
| Cosmetics | Hair styling aids<br>Alcohol-based preparations<br>Perfumes and colognes<br>Emulsion creams, lotions, and shampoos | <br>Alcohol-soluble thickener and film-former<br><br>Emulsion stabilizer, thickener |
| Encapsulation | Micro- and macroencapsulation | Soluble, edible, flexible film barrier, fast release |
| Extrusion | Film and sheet<br>Profiles and filament | Binder, thermoplastic, water- and<br>solvent-soluble |
| Foods | Whipped toppings<br>Edible coatings for nuts and candies<br>Glaze for confections<br>Fabricated foods | Stabilizer, whipping aid<br>Protective coating and oil barrier<br>High gloss and color coatings<br>Binder for molding and extrusion |
| Molding | Injection-, compression-, and blow-molding | Binder, thermoplastic, water- and<br>solvent-soluble |
| Paint removers | Acid-based<br>Scrape-off and flush-off | Thickener, acid resistant |
| Paper | Coatings | Solvent-soluble, flexible, thermoplastic film barrier |
| Pharmaceuticals | Tablet binder<br>Tablet coating<br>Modified release<br>Liquids and semisolids | Aqueous and solvent solubility<br>Thermoplastic binder<br>Non-ionic, pH insensitive<br>Thickener, suspending agent, diffusion barrier<br>Flexible films, surface active |
| Plastic foams | Foamed sheet, tube, rod | Thermoplasticity |
| Polyvinyl chloride processing | Suspension polymerization | Surface-active protective colloid |
| Printing | Inks (water-, alcohol-, and glycol-based) | Thickener, binder, suspending agent |
| Miscellaneous | Cleaners (acid-based)<br>Polishes (aqueous- and solvent-based) | Thickener, acid resistant<br>Thickener, stabilizer, suspending agent |

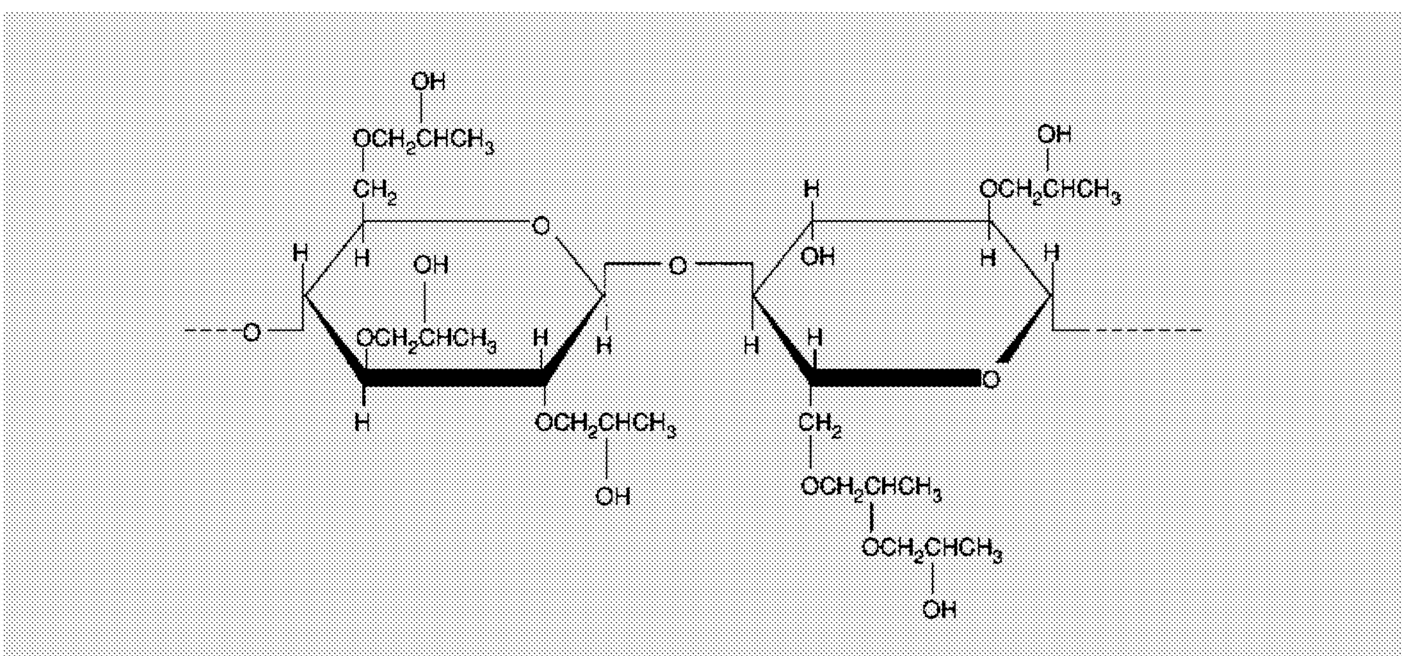
# CHEMISTRY

KLUCEL® hydroxypropylcellulose is manufactured by reacting alkali cellulose with propylene oxide at elevated temperatures and pressures. The propylene oxide can be substituted on the cellulose through an ether linkage at the three reactive hydroxyls present on each anhydroglucose monomer unit of the cellulose chain. Published information suggests that etherification takes place in such a way that hydroxypropyl substituent groups contain almost entirely secondary hydroxyls. The secondary hydroxyl present in a side chain is available for further reaction with the oxide, and chaining-out may take place. This results in formation of side chains containing more than one mole of combined propylene oxide.

It is probable that most of the primary hydroxyls on the cellulose have been substituted and that the reactive groups remaining are secondary hydroxyls. Some typical molecular weight values are given in Table I.

An idealized structure for a portion of a hydroxypropyl-cellulose molecule with a molar substitution (MS) of 3.0 is given in Figure 1.

**Figure 1**
**Structure of Hydroxypropylcellulose (MS 3.0)**



# GRADES AND VISCOSITY TYPES

KLUCEL® hydroxypropylcellulose is produced in several grades, determined by intended markets. For each grade, up to six viscosity types are available designated as H, M, G, J, L, E. Product designation is a combination of viscosity type followed by grade designation. For example: Klucel H Industrial, HF, H CS, and HF Pharm. Table I gives the water and ethanol viscosity specifications by grade for each available viscosity type.

Grades of KLUCEL

| Intended Market | Grade Designation |
|---|---|
| Industrial | Industrial |
| Food | F |
| Personal Care | CS |
| Pharmaceutical | F Pharm |

Table I – KLUCEL Viscosity Types, Viscosities (cps)[a], and Their Corresponding Molecular Weights

| Industrial Grade | Concentration in Water by Weight, % | | | | |
|---|---|---|---|---|---|
| Viscosity Types | 1 | 2 | 5 | 10 | Mw[b] |
| H Industrial | 1,275-3,500 | – | – | – | 1,150,000 |
| M Industrial | – | 3,500-7,500 | – | – | 850,000 |
| G Industrial | – | 125- 450 | – | – | 370,000 |
| J Industrial | – | – | 125-450 | – | 140,000 |
| L Industrial | – | – | 65-175 | – | 95,000 |
| E Industrial | – | – | – | 250-800 | 80,000 |
| **Food Grade, Personal Care Grade, Pharmaceutical Grade** | | | | | |
| Viscosity Types | 1 | 2 | 5 | 10 | Mw[b] |
| H CS, HF Pharm | 1,500-3,000 | – | – | – | 1,150,000 |
| M CS, MF Pharm | – | 4,000-6,500 | – | – | 850,000 |
| GF, G CS, GF Pharm | – | 150- 400 | – | – | 370,000 |
| JF, J CS, JF Pharm | – | – | 150-400 | – | 140,000 |
| LF, L CS, LF Pharm | – | – | 75-150 | – | 95,000 |
| EF, E CS | – | – | – | 200-600 | 80,000 |
| EF Pharm | – | – | – | 300-600 | 80,000 |

| Personal Care Grade, Pharmaceutical Grade | Concentration in Anhydrous Ethanol, by Weight, % | | | | |
|---|---|---|---|---|---|
| Viscosity Types | 1 | 2 | 5 | 10 | Mw[b] |
| H CS, HF Pharm | 1,000-4,000 | – | – | – | 1,150,000 |
| M CS, MF Pharm | – | 3,000-6,500 | – | – | 850,000 |
| G CS, GF Pharm | – | 75- 400 | – | – | 370,000 |
| J CS, JF Pharm | – | – | 75-400 | – | 140,000 |
| L CS, LF Pharm | – | – | 25-150 | – | 95,000 |
| E CS, EF Pharm | – | – | – | 150-700 | 80,000 |

[a] All viscosities are determined at 25°C using a Brookfield LVF viscometer with spindle and speed combinations depending on viscosity level. See Appendix.
[b] Weight-average molecular weight determined by size exclusion chromatography.

# PROPERTIES

All grades conform to the specifications given in Table II. Typical properties are given in Table III.

Table II – Product Specifications for KLUCEL® HPC

| | |
|---|---|
| Viscosity | Values are shown in Table I. |
| Physical form | White to off-white, granular solid |
| Particle size[1] | min. 85% through 30 mesh<br>min. 99% through 20 mesh<br>Industrial Grade:<br>min. 80% through 30 mesh<br>min. 98% through 20 mesh |
| Ash content, calculated as $Na_2SO_4$, % | 0.2 max, silica free (No ash specification for Industrial Grades) |
| Moisture content (as packed), % | 5.0 max |
| pH in water solution | Food and Personal Care Grades: 5.0-8.0<br>Pharmaceutical Grade: 5.0-7.5 |
| Moles of substitution | 3.4 to 4.4 |

[1]Fine x-grind Pharmaceutical Grade E and H viscosity types are available and designated as EXAF Pharm and HXAF Pharm. Particle size specifications are:
min. 80% through 100 mesh
min. 90% through 80 mesh
min. 99.9% through 60 mesh

## MOISTURE ABSORPTION

KLUCEL absorbs moisture from the atmosphere, as do other water-soluble materials. The amount absorbed depends on relative humidity and temperature of the environment. As packed, moisture content of all grades does not exceed 5% by weight, and is generally between 2% and 3%. It is suggested that KLUCEL be stored in tightly closed containers and in a dry atmosphere to prevent any increase in moisture content.

KLUCEL has a low affinity for water. At any given relative humidity (RH), it has a lower equilibrium moisture content than most other water-soluble polymers. Typical values for KLUCEL are given below.

| | |
|---|---|
| Equilibrium moisture content at 50% RH and 73°F (23°C) | 4% |
| Equilibrium moisture content at 84% RH and 73°F (23°C) | 12% |

## POLYMER STABILITY

Long-term storage stability of KLUCEL hydroxypropylcellulose is affected by the initial molecular weight of the polymer and storage conditions. Studies have shown that low- and medium-viscosity types maintain an average of 97% of their original viscosity after three years when stored at room temperature with frequent exposure to the atmosphere.

H viscosity types have a higher molecular weight and, therefore, are more susceptible to viscosity loss over time. Studies have shown that H viscosity types may sustain up to 10% viscosity loss after one year and 20% viscosity loss after two years. Storage at room temperatures with infrequent exposure to the atmosphere improves viscosity stability. Customers are encouraged to retest H viscosity types after one year and quarterly thereafter to assure material viscosity.

Table III – Typical Properties for KLUCEL

| | |
|---|---|
| **Product as Shipped** | |
| Solubility in water[a] | Solutions are clear and smooth at temperatures below 38°C. |
| Solubility in organic solvents | Dissolves easily in many polar solvents to give clear, smooth solutions |
| Bulk density, g/ml | 0.5 (varies with type) |
| Softening temperature | 100-150°C |
| Burnout temperature | Burns out completely at 450° to 500°C in $N_2$ or $O_2$ |
| Biological Oxygen Demand | 14,000 ppm |
| **Solutions in Water** | |
| Specific gravity, 2% solution at 30°C | 1.010 |
| Refractive index, 2% solution | 1.337 |
| Surface tension, at 0.1% | 43.6 dynes/cm |
| Interfacial tension, 0.1% KLUCEL in water vs refined mineral oil | 12.5 dynes/cm |
| Bulking value in solution, | 0.04 (0.334) gal/lb (l/kg) |

[a]Silicon dioxide is added as an anticaking agent and may contribute a slight haze.

## BURNOUT TEMPERATURE

KLUCEL has excellent binding properties. It is often used as a temporary binder in production of ceramics, glazes, refractories, and powdered metal products. This polymer is vaporized, or burned out, over the temperature range of 250° to 500°C in oxidizing, reducing, or inert atmospheres. The very low ash content of the original KLUCEL and the complete absence of organic residues after firing ensure virtually uncontaminated end products after burnout.

# DISPERSION AND DISSOLUTION

KLUCEL® HPC is soluble in water at room temperature. It is insoluble in water above 45°C. It is readily soluble in many organic solvents, hot or cold. The best methods for preparing solutions of KLUCEL in water or organic solvents are described in the following paragraphs.

Note: As a general aid to preparation of solutions, the following points should be kept in mind:

- Wherever possible, KLUCEL should be put into solution before adding other soluble ingredients. Other dissolved materials compete for the solvent and slow the solution rate of KLUCEL. In this regard, soft water is preferred to hard water for solution preparation.
- KLUCEL is less soluble in hot water than in cold. In organic solvents, application of heat speeds the solution rate.

**IN WATER**

Most soluble polymers have a tendency to agglomerate, or lump, when the dry powder is first wet with solvent. Hydration of the outer surface of a particle, or an agglomerate of particles (lump), results in the formation of a viscous gel layer that inhibits wetting of the inside materials. The faster the rate of hydration of the polymer, the more quickly the gel layer will be developed, and the greater the tendency for lumping as the dry powder is added to the solvent.

KLUCEL hydrates somewhat slowly, and lumping can be avoided during solution preparation if the recommended procedures are followed. Lump formation should be avoided, as this can greatly increase the time required to prepare homogeneous solutions.

To prepare lump-free, clear solutions of KLUCEL in the shortest time, the following methods are suggested.

**Method 1**

The preferred method involves pre-slurrying the powder in a nonsolvent, such as hot water or glycerin, prior to addition to the main volume of water.

In the first step, prepare a high-solids slurry by adding dry KLUCEL powder to 6 times (or more) its weight of well-agitated hot water at a temperature of 50° to 60°C. Temperature should not exceed the 60°C maximum indicated. The hot slurry must be maintained above 50°C during this presoak to ensure that there is no premature dissolving of the particles that would result in the formation of a gelatinous mass. The slurry should be allowed to stir for a few minutes before addition to the main volume of cold water. This presoak results in a faster dissolving of particles in the second step.

In the second step, the hot slurry is diluted with cold water (room temperature or lower). Agitation is continued until all particles are dissolved and solution is completely free of gels. High-shear agitation is not necessary, and may be undesirable because of the tendency for foaming and air entrainment. In this dissolving step, the time factor is more important than high shear when it comes to ensuring complete solution of all gel particles.

Dissolving periods of 10 minutes or more may be required, depending on solution concentration and viscosity type being used. Solutions of lower-viscosity KLUCEL types at low-solids concentration require the shortest time for preparation.

**Method 2**

Add powdered KLUCEL to the vortex of well-agitated water at room temperature. The rate of addition must be slow enough to permit particles to separate in the water. Addition of the powder should be completed, however, before any appreciable viscosity buildup is obtained in the solution. The rate of agitation then may be reduced, but continued until a gel-free solution is obtained. Throughout the mixing period, solution temperature should be maintained below 35°C.

**Method 3**

Dry-blend KLUCEL with any inert or nonpolymeric soluble material that will be used in the formulation. Blending aids separation of particles of KLUCEL at first wetting and reduces the tendency to lump. For best results, KLUCEL should be less than 20% of the total dry blend. This blend is then handled as described in Method 2.

**IN ORGANIC LIQUIDS**

All types of KLUCEL have excellent solubility in a wide range of polar organic liquids and give clear, smooth solutions at ambient or elevated temperatures. There is no tendency for precipitation of KLUCEL in hot organic solvents; this is in contrast to its behavior in water solutions. Generally, the more polar the liquid, the better the solution. Methyl and ethyl alcohol, propylene glycol, dioxane, and Cellosolve are some of the best organic solvents for all types of KLUCEL.

Table VIII, page 15, lists the type of solutions obtained with G viscosity types in many organic liquids. The molecular weight of the type of KLUCEL can have a marked effect on solution quality in an organic liquid that is a borderline solvent for KLUCEL.

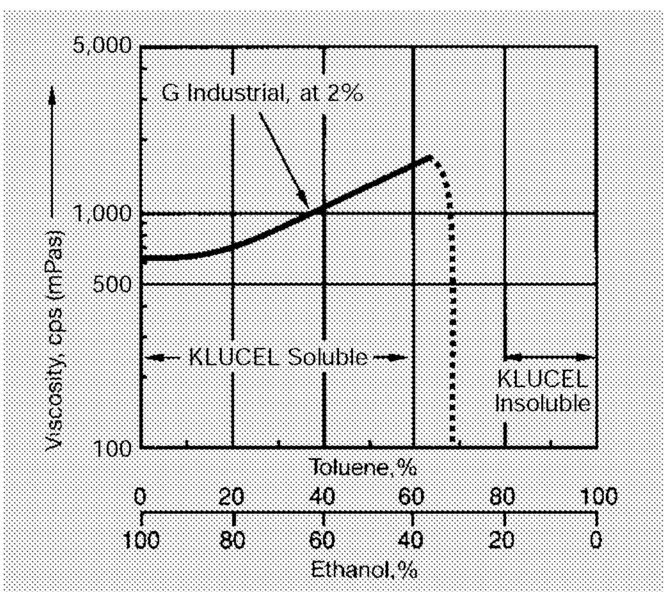
G viscosity types are intermediate in viscosity (i.e., molecular weight) between high H viscosity types and very low E viscosity types. In comparison with G viscosity types, lower-viscosity types are more readily soluble and may give better solutions. The higher-viscosity types may give slightly inferior solutions in some of the liquids listed in the table. For example, acetone gives excellent solutions with the E viscosity types, but acetone solutions of the G viscosity types are hazy and somewhat granular.

Solution quality in borderline solvents often can be greatly improved through the use of small quantities of cosolvents. Water, methanol, and ethanol function as excellent cosolvents and, in many cases, are effective in relatively small quantities (5% to 15%). For example, methylene chloride is a borderline solvent for high H viscosity types, and solutions are granular. Addition to the system of 10% methanol results in a smooth solution of normal viscosity (5,000 cps at 1% solids).

Elevated temperatures will improve solvent power of organic liquids for KLUCEL® HPC, and there is no tendency for precipitation of the polymer at these elevated temperatures. Heating of solvents will (a) reduce viscosity, (b) increase rate of solution, and (c) improve solution quality in the case of borderline solvents.

Aliphatic and aromatic hydrocarbons and petroleum distillates are nonsolvents for KLUCEL. However, relatively large quantities of these nonsolvents can be tolerated in a solution if KLUCEL is first dissolved in a solvent that is miscible with these nonsolvents. Examples of solvent-nonsolvent systems are given in Table VIII. Figure 2 details the effect of solvent composition on viscosity of a solution of G Industrial in a toluene:ethanol system.

**Figure 2**
**Viscosity of G Industrial Dissolved in Toluene-Ethanol**




In general, principles discussed for preparing water solutions apply when using organic solvents to make solutions of KLUCEL. Methods 1, 2, and 3 described for preparing water solutions can therefore be used to prepare solutions of KLUCEL in organic solvents. The "pre-slurry" principle of Method 1 can be employed through the use of nonsolvents such as glycerin, aliphatics, aromatics, and others.

In aqueous-organic systems, the proportion of organic solvent will determine whether elevated temperature will speed or slow the rate of solution of KLUCEL. (See Viscosity and Precipitation Temperature in Aqueous Alcohols, page 17).

**IN HOT-MELTS AND WAXES**

At elevated temperatures, many waxes are sufficiently good solvents so that KLUCEL is readily dissolved by addition of the dry powder to the stirred molten wax. Examples of materials that are good solvents for KLUCEL at an elevated temperature are: acetylated monoglycerides (e.g., Myvacet 5-00 and 7-00 series); glycerides (e.g., Myverol 18-07); polyethylene glycols (e.g., Carbowax); polypropylene glycol, pine oil, and tall oil fatty acids.

KLUCEL is compatible with a number of high-molecular-weight, high-boiling waxes and oils, and can be used to modify the properties of these materials. The addition of KLUCEL to these systems will increase viscosity and improve hardness and crack resistance to coatings.

# PROPERTIES OF SOLUTIONS

KLUCEL® HPC has excellent solubility in water and in many polar organic solvents, as discussed in the Dispersion and Dissolution section starting on page 7. Solutions are clear, smooth, and exceptionally free from gels and fibers. Solutions are non-Newtonian in flow, since they change in viscosity with rate of shear. But solutions display little or no thixotropy.

Because KLUCEL is used extensively to modify viscosity of solutions, dispersions, emulsions, and suspensions involving water and/or organic solvents, a discussion of some of the factors that affect solution viscosity follows.

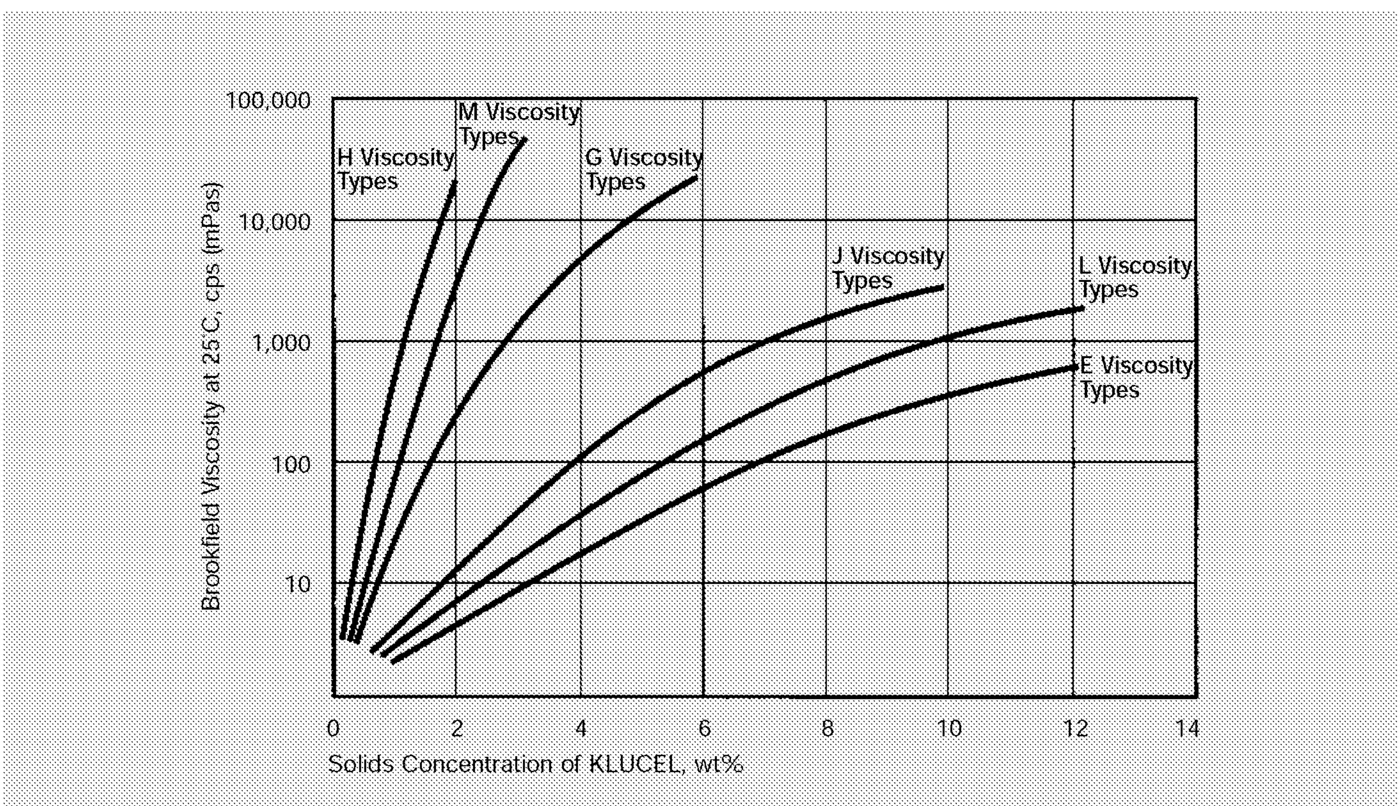
## AQUEOUS SOLUTIONS

At room temperature, solutions of KLUCEL can be prepared in a wide range of viscosities, depending on concentration and viscosity type used. Since solutions are non-Newtonian, it is essential to standardize viscosity determination methods. The method used in the control laboratory at Aqualon is described in detail in the Appendix.

### Effect of Concentration and Viscosity Type

The viscosity of solutions of KLUCEL increases rapidly with concentration and becomes almost a straight-line relationship when plotted on a semi-log basis. (See Figure 3.) The bands in this figure indicate the viscosity range within which each type is supplied. (See also Table I, page 5.)

**Figure 3**
**Effect of Concentration and Type of KLUCEL on Viscosity of Water Solutions**



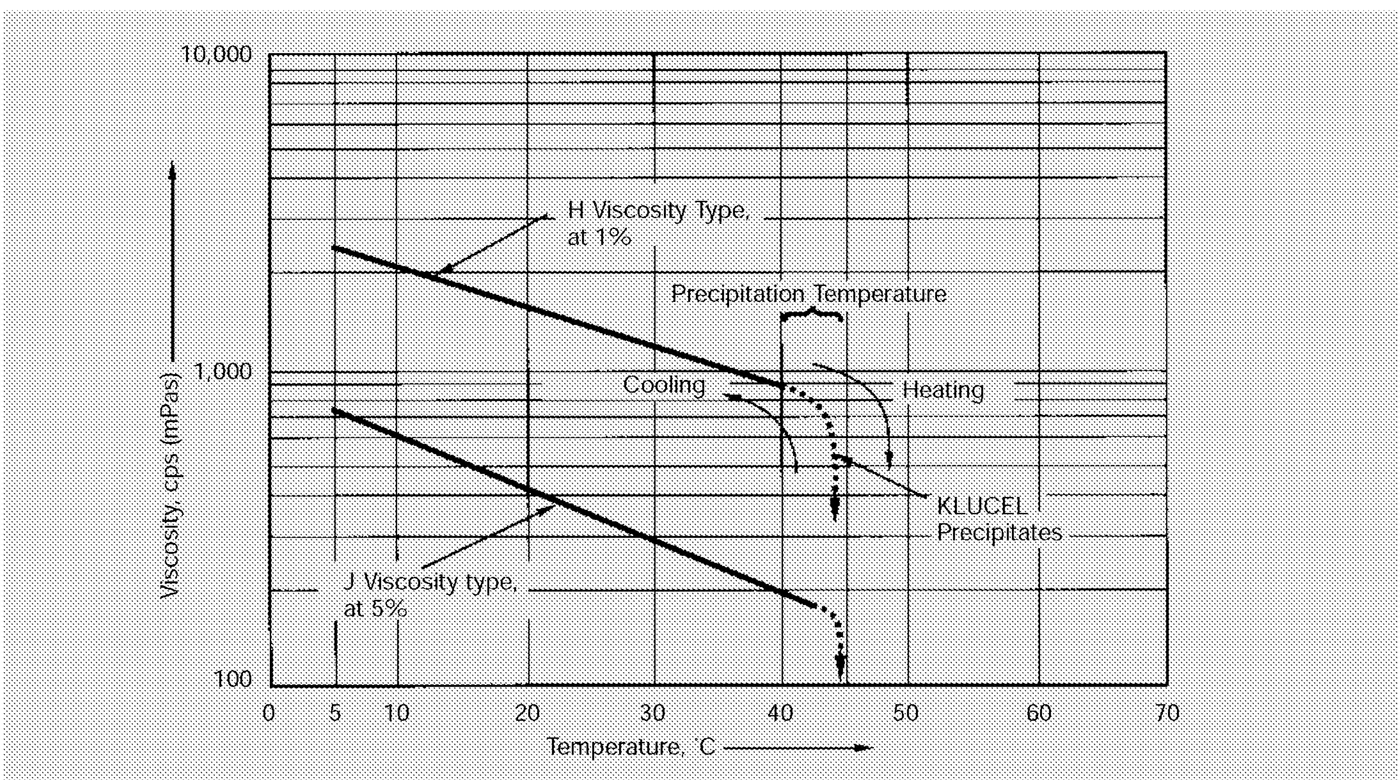
## Rheology

Solutions of KLUCEL are exceptionally smooth-flowing and exhibit little or no structure or thixotropy. However, solutions are pseudoplastic under conditions of high rates of shear and will show a temporary decrease in viscosity while under shear. The viscosity returns to the original value when the shear is removed. The lower the molecular weight of KLUCEL and the lower the shear rate, the less will be this decrease in viscosity experienced under shear.

## Effect of Temperature

Viscosity of an aqueous solution of KLUCEL decreases as temperature is increased. This effect is normal for polymers in solution. This change in viscosity is illustrated in Figure 4 for H viscosity type and J viscosity type. As shown, viscosity is halved as temperature is raised through 15°C. This effect is uniform up to the precipitation temperature (40° to 45°C).

**Figure 4**
**Effect of Temperature on the Viscosity of Aqueous Solutions of KLUCEL**



## Precipitation Temperature in Water

As already stated, KLUCEL® HPC will precipitate from water solution at a temperature between 40° and 45°C. This precipitation is completely reversible. The polymer redissolves upon cooling the system below 40°C with stirring, and the original viscosity is restored.

As the temperature reaches 40° to 45°C, this precipitation phenomenon is evidenced by appearance of cloudiness in the aqueous solution and by a marked reduction in viscosity. These effects are due to separation of the polymer as a highly swollen precipitate. The transition from dissolved to precipitated polymer takes place without the formation of a gel. The only apparent viscosity change is one of a rapid decrease, as shown in Figure 4.

The form in which KLUCEL precipitates from aqueous solutions depends not only on the molecular weight of the polymer, but also on other materials present in solution and whether or not stirring is employed. Low-viscosity types tend to separate as highly swollen and finely divided precipitates. High-viscosity types, particularly under agitation, may agglomerate on heating and form a stringy, rather than a finely divided, precipitate. The addition of Aqualon® cellulose gum or surfactants reduces the tendency for agglomeration of the KLUCEL polymer as it precipitates, and by this means, the high-viscosity types also can be separated in finely divided form.

The precipitation temperature of KLUCEL is increased through the addition of organic liquids that are solvents for the polymer. The precipitation temperature in aqueous alcohols and aqueous glycol is discussed in another section.

The precipitation temperature is lower in the presence of relatively high concentrations of other dissolved materials that compete for the water in the system. The magnitude of the lowering is dependent on the nature and concentration of the other dissolved ingredients. The data in Table IV illustrate this effect.

Table IV – Comparative Effect of Solution Composition on Precipitation Temperature

| Ingredients and Concentration | Precipitation Temperature, °C |
|---|---|
| 1% H viscosity type | 41 |
| 1% H viscosity type + 1.0% NaCl | 38 |
| 1% H viscosity type + 5.0% NaCl | 30 |
| 0.5% H viscosity type + 10% Sucrose | 41 |
| 0.5% H viscosity type + 20% Sucrose | 36 |
| 0.5% H viscosity type + 30% Sucrose | 32 |
| 0.5% H viscosity type + 40% Sucrose | 20 |
| 0.5% H viscosity type + 50% Sucrose | 7 |

## Compatibility With Surfactants

Compatibility of KLUCEL with surface-active agents will vary according to the particular agent and concentration, as well as to the grade and concentration of KLUCEL used.

Because of its hydroxypropyl substitution, KLUCEL is more lipophilic in nature than other water-soluble cellulose derivatives. Accordingly, it is compatible with a wide range of anionic, nonionic, cationic, and amphoteric surfactants.

## Addition of Certain Ionic Surfactants

Studies with KLUCEL using certain ionic surfactants have aided the development of technology to permit thickening at temperatures in excess of the normal cloudpoints for KLUCEL. Aqueous solutions of M viscosity type and sodium lauryl sulfate, at a surfactant to KLUCEL ratio of 1:3 or greater, result in cloudpoints in excess of 70°C. At some ratios, cloudpoints greater than 95°C may be achieved.

Included in these studies were the following ionic surfactants, which proved to be effective:

- Sodium lauryl sulfates
- Ammonium lauryl sulfates
- Lauryl alcohol ether sulfate
- Trimethylcoco ammonium chloride

The nonionic surfactants studied were not effective in raising the cloudpoints.

## Effect of pH

KLUCEL HPC is a nonionic polymer, and viscosity of water solutions is not affected by changes in pH. The viscosity of solutions remains unchanged as pH is varied over the range of 2 to 11.

However, where long-term storage stability is required, the solution pH is an important consideration because of degradation that can occur under highly acid or alkaline conditions, as described in the section titled Viscosity Stability, page 13.

## Effect of Inorganic Salts

The compatibility of KLUCEL with dissolved inorganic salts in water solution varies according to the salt. If relatively high concentrations of dissolved salts are used, there is a tendency for the KLUCEL polymer to be "salted out" from solution as a finely divided and highly swollen precipitate.

This salting-out phenomenon generally results in some decrease in viscosity and in the appearance of cloudiness in the solution. In borderline cases, this salting out may not be immediately apparent, but may occur upon standing.

The compatibility of G viscosity types with a number of selected salts is illustrated in Table V.

Table V – Compatibility of KLUCEL® HPC With Some Inorganic Salts[a]

| Salt | Salt Concentration, % by weight | | | |
|---|---|---|---|---|
| | 2 | 5 | 10 | 50 |
| Aluminum sulfate | C | I | – | – |
| Ammonium nitrate | C | C | C | I |
| Ammonium sulfate | C | I | – | – |
| Calcium chloride | C | C | C | I |
| Disodium phosphate | I | – | – | – |
| Ferric chloride | C | C | C | I |
| Potassium ferrocyanide | C | C | I | – |
| Silver nitrate | C | C | C | I |
| Sodium acetate | C | C | I | – |
| Sodium carbonate | C | I | – | – |
| Sodium chloride | C | C | I | – |
| Sodium nitrate | C | C | C | I |
| Sodium sulfate | C | I | – | – |
| Sodium sulfite | C | I | – | – |
| Sodium thiosulfate | C | I | – | – |
| Sucrose | C | C | C | I |

Key: C = compatible; I = incompatible

[a] Tests were conducted by adding a 2% solution of G viscosity type to salt solutions of various concentrations. The salt concentration in the system is indicated, and the final concentration of KLUCEL was approximately 0.1% by weight in all cases.

**Compatibility With Other Polymers**

KLUCEL has a wide range of compatibility with organic materials. The dual solubility of KLUCEL permits its admixture with water-soluble, as well as solvent-soluble, resins, polymers, and organic liquids.

In spite of this wide compatibility, the KLUCEL polymer, when used in aqueous systems, may not tolerate high concentrations of other dissolved materials. The balance of hydrophilic-lipophilic properties of the polymer, which are required for dual solubility, reduces its ability to remain hydrated in the presence of high concentrations of other dissolved materials. KLUCEL may precipitate or "salt out" under these conditions.

A detailed discussion of compatibility with natural and synthetic polymers and inorganic salts follows.

*Water-Soluble Polymers*

KLUCEL is compatible with most natural gums and synthetic water-soluble polymers. Solutions in water are homogeneous, and films cast from these solutions are uniform. The following have been tested and found to be compatible:

- AQUALON® sodium carboxymethylcellulose (CMC)
- NATROSOL® hydroxyethylcellulose (HEC)
- BENECEL® methylcellulose (MC)
- SUPERCOL® guar gum
- Gelatin
- Sodium caseinate
- Polyethylene oxide
- Carbowax 1000
- Polyvinyl alcohol
- Sodium alginate
- Locust bean gum

The effect on solution viscosity of blends of KLUCEL and other water-soluble polymers varies, depending on the ionic nature of the copolymer. To illustrate, blends of KLUCEL with NATROSOL hydroxyethylcellulose and CMC were studied. The blends were prepared at a 1:1 ratio of the two polymers. The results of this study are given in Table VI.

Solution viscosity of blends of nonionic polymers KLUCEL and NATROSOL was essentially in agreement with the calculated value. This was true for all viscosity types studied.

When solutions of blends of KLUCEL and an ionic polymer, CMC, were prepared, the resultant viscosity was greater than the calculated value. The synergism of this combination increased with increasing molecular weight of the polymers.

The data shown in Table VI were obtained with solutions prepared in distilled water or tapwater. The synergistic effect may be drastically reduced in the presence of low levels of dissolved salts or if pH is below 3 and above 10.

Table VI – Blends of KLUCEL® HPC and Other Water-Soluble Polymers: Effect on Viscosity

| Polymer Blend (1:1) | Concentration, % | Solution Viscosity, cps (mPas) | | |
|---|---|---|---|---|
| Viscosity Types | | Expected | Initial | After 24 Hours |
| J and NATROSOL® 250J HEC | 5 | 235 | 240 | 235 |
| M and NATROSOL 250M | 2 | 6,250 | 5,900 | 5,600 |
| H and NATROSOL 250H | 1 | 2,320 | 2,440 | 2,440 |
| H and AQUALON® CMC-7H | 1 | 2,220 | 4,400 | 3,860 |

*Water-Insoluble Polymers*
KLUCEL is compatible with many natural and synthetic latexes available as emulsions in water. KLUCEL is soluble in the aqueous phase, and uniform films and coatings are obtained upon drying.

Using common solvents, KLUCEL has been incorporated with water-insoluble polymers such as zein, shellac, AQUALON ethylcellulose, and cellulose acetate phthalate. Films and coatings prepared from these systems are homogeneous and of good quality.

**Viscosity Stability**
Water solutions of KLUCEL possess best viscosity stability when pH is held between 6.0 and 8.0, and when the solutions are protected from light, heat, and action of microorganisms.

KLUCEL in water solution, like other water-soluble polymers, is susceptible to both chemical and biological degradation. This degradation generally results in reduction of molecular weight of the dissolved polymer, with an accompanying decrease in viscosity of the solution. Some loss of solution clarity may occur in cases of severe biological degradation.

KLUCEL has demonstrated greater resistance to chemical and biological degradation than other cellulose ethers. Techniques to minimize degradation mechanisms are discussed in the following two sections.

*Hydrolysis and Oxidation*
KLUCEL in solution is susceptible to acid hydrolysis, which causes chain scission and loss of viscosity of the solution. The rate of hydrolysis increases with temperature and hydrogen ion concentration. Solutions should be buffered to pH 6.0 to 8.0 and maintained at low temperature to minimize acid hydrolysis.

Alkali-catalyzed oxidative degradation will also degrade the polymer and result in decrease in viscosity of solution. The degradation can result from presence of dissolved oxygen or oxidizing agents in the solution. Peroxides and sodium hypochlorite under alkaline conditions cause rapid degradation. For best stability on storage, pH should be maintained between 6.0 and 8.0 and antioxidants should be used if oxidative degradation is likely to occur.

Ultraviolet light will degrade the cellulose, and solutions of KLUCEL will undergo some decrease in viscosity if exposed to light for several months.

*Biological Stability*
The high level of substitution of KLUCEL improves resistance of this polymer to degradation by cellulase enzymes produced by molds and bacteria. However, water solutions are susceptible to degradation under severe condtions, and a viscosity decrease may result. If prolonged storage is contemplated, a preservative is recommended.

Certain enzymes, produced by microbial action, will degrade KLUCEL in solution. If microbial contamination is present in makeup water, it is important that sterilization techniques effective against enzymes as well as against microorganisms be employed prior to preparing the solution of KLUCEL.

Solutions of KLUCEL in organic solvents do not generally require preservative.

**Preservatives**
Listed below are some of the preservatives that are effective in preserving solutions of KLUCEL. It is recommended that the preservative manufacturer be consulted regarding kind, amount, and rate of use for the preservative to be added.

- Dowicil 100 n-(3-chloroallyl)-hexaminium chloride
- Formaldehyde
- Phenol
- Omadine

Some of the preservatives that are effective with KLUCEL and are in compliance with the Food and Drug Administration for use in food are listed below.

- Sodium benzoate
- Sorbic acid and its potassium, sodium, and calcium salts
- Sodium propionate
- Methyl and propyl parahydroxybenzoate

Note: Solutions of KLUCEL have demonstrated some incompatibility with a number of preservatives based on substituted phenol derivatives.

**Surface and Interfacial Tension**

KLUCEL® HPC is a surface-active polymer. Water solutions display greatly reduced surface and interfacial tension. Because of this, KLUCEL functions as an aid in both emulsifying and whipping. These properties, coupled with protective colloid action, enable it to perform dual functions in the following systems:

- Oil-in-Water Emulsions – Stabilizer and emulsification aid
- Foamed Systems – Stabilizer and whipping aid

The reduction in surface and interfacial tension of water solutions containing KLUCEL is illustrated in Table VII. All viscosity types have essentially the same effects, and a concentration of KLUCEL as low as 0.01% produces close to the maximum reduction in surface tension.

*Recommended Defoamers*

The low surface tension of water solutions containing KLUCEL tends to promote foaming and air entrainment. If this presents a difficulty, a water-dispersible antifoam agent can be used and should be added to the water prior to solution preparation.

Water-dispersible defoamers such as HERCULES® 1512 defoamer; Colloid 581-B; Nopco NDW and KFS; Antifoam AF; or lauryl or octyl alcohol are effective. Defoamed concentrations generally run 25 to 200 ppm, but it is suggested that the manufacturers be consulted for their recommendations for the particular system involved.

Table VII – Surface and Interfacial Tensions of KLUCEL Solutions at 25°C

| KLUCEL, wt% | Surface Tension, dynes/cm (mN/m) | Interfacial Tension vs Refined Mineral Oil, dynes/cm (mN/M) |
|---|---|---|
| 0 (water) | 74.1 | 31.6 |
| 0.01 | 45.0 | – |
| 0.1 | 43.6 | 12.5 |
| 0.2 | 43.0 | – |

## VISCOSITY IN ORGANIC SOLVENT SOLUTIONS

The viscosity-vs-concentration curve for KLUCEL® HPC dissolved in organic liquids, which are good solvents for the polymer, shows the same general pattern as that for KLUCEL dissolved in water (Figure 3, page 9). The viscosity rises rapidly as the concentration of polymer is increased. The curves for viscosity in ethanol and methanol parallel those for viscosity in water, but are displaced toward somewhat lower viscosity values.

Table VIII – Solvents for KLUCEL[a]

| | |
|---|---|
| **A. CLEAR AND SMOOTH** | |
| Acetic acid (glacial) | Isopropyl alcohol (95%) |
| Acetone:water (9:1) | Methanol |
| Benzene:methanol (1:1) | Methyl Cellosolve |
| Cellosolve | Methylene chloride: methanol (9:1) |
| Chloroform | Morpholine |
| Cyclohexanone | M-Pryol |
| Dimethyl formamide | Propylene glycol |
| Dimethyl sulfoxide | Pyridine |
| Dioxane | t-Butanol:water (9:1) |
| Ethyl alcohol | Tetrahydrofuran |
| Ethylene chlorohydrin | Toluene:ethanol (3:2) |
| Formic acid (88%) | Water |
| Glycerin:water (3:7) | |
| **B. MODERATELY GRANULAR AND/OR HAZY** | |
| Acetone | Methyl acetate |
| Butyl acetate | Methyl ethyl ketone |
| Butyl Cellosolve | Methylene chloride |
| Cyclohexanol | Naphtha:ethanol (1:1) |
| Isopropyl alcohol (99%) | Tertiary butanol |
| Lactic acid | Xylene:isopropyl alcohol (1:3) |
| **C. INSOLUBLE** | |
| Aliphatic hydrocarbons | Methyl chloroform |
| Benzene | Mineral oils |
| Carbon tetrachloride | Soybean oil |
| Dichlorobenzene | Toluene |
| Kerosene | Gasoline |
| Trichloroethylene | Glycerin |
| Xylene | Linseed oil |

[a] Solvents were tested using G viscosity types at 2% solids concentration by weight. All ratios indicated in this table are on a by-weight basis.

Where organic liquids that are borderline solvents for KLUCEL® HPC are used, unusual viscosity effects can be observed. Viscosity may be abnormally high or abnormally low, depending on the degree of solvation of the polymer. For example, as shown in Table IX, H viscosity types in methylene chloride gave a poor solution with reduced viscosity. The L viscosity types, which was better solvated, gave a solution with unusually high viscosity. In both cases, the addition of a small amount of a cosolvent (10% methanol) gave solutions with normal viscosities.

Table IX lists typical solution viscosities for various types of KLUCEL in a number of solvents.

Table IX – Comparative Viscosity of KLUCEL in Water and Certain Organic Solvents

| Solvent | Viscosity[a] Type of KLUCEL and Concentration, cps (mPas) | | | |
|---|---|---|---|---|
| | H at 1% | G at 2% | L at 9% | E at 10% |
| Water | 2,100 | 270 | 80 | 275 |
| Methanol | 800 | 85 | 25 | 75 |
| Methanol:water (3:7 by weight) | – | 360 | – | – |
| Ethanol | 1,600 | 210 | 65 | 255 |
| Ethanol:water (3:7) | – | 500 | – | – |
| Isopropyl alcohol (99%) | [b] | [b] | 145 | 570 |
| Isopropyl alcohol (95%) | – | – | 130 | 420 |
| Acetone | [b] | [b] | 50 | 175 |
| Methylene chloride | 4,500[b] | – | 1,240[b] | 14,600[b] |
| Methylene chloride:methanol (9:1) | 5,000 | – | 400 | – |
| Chloroform | – | – | 2,560[b] | 17,000[b] |
| Propylene glycol | 6,000 | 6,640 | 5,020 | >10,000 |
| Ethylene chlorohydrin | 470 | 430 | 310 | 1,110 |

[a] Viscosities shown are presented only as typical values. Some variation in these viscosities will be obtained from lot to lot of each type of KLUCEL.
[b] Borderline solvent for the particular type of KLUCEL. Solutions are granular and may be hazy.

**Viscosity and Precipitation Temperature in Aqueous Alcohols**

The viscosity of solutions of KLUCEL® HPC in aqueous alcohols varies with composition of the solvent. The viscosity goes through a maximum value at a solvent composition of 7 parts water: 3 parts alcohol by weight. This is illustrated in Figure 5. This type of viscosity curve is obtained when KLUCEL is added directly to aqueous alcohol or when it is first dissolved in either water or alcohol with subsequent addition of the second solvent.

Addition of alcohol to a water solution of KLUCEL will increase the temperature at which the polymer will precipitate from solution. Temperature elevation is dependent on type and concentration of alcohol. The effect obtained with methanol and ethanol is detailed in Figure 6. As shown, solutions of KLUCEL containing 45% (by volume) of ethanol or methanol can be heated to the boiling point of the solution without precipitation of KLUCEL.

Propylene glycol performs similarly to methanol, and elevation of precipitation temperature falls on the same curve. Other water-miscible organic liquids, which are good solvents for KLUCEL, will also function to elevate precipitation temperature of the polymer in the system.

**Figure 5**
**Viscosity of Aqueous Alcohol Solutions (G viscosity types at 2% concentration by weight)**

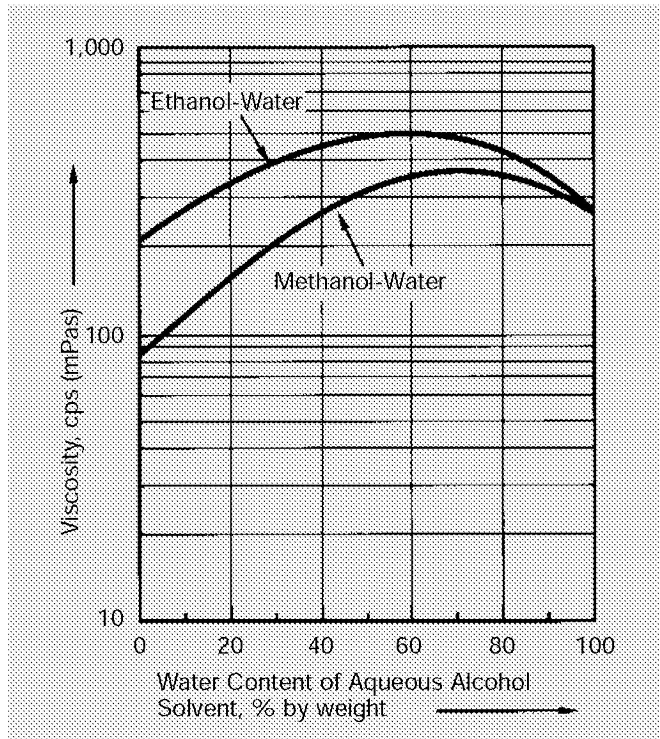
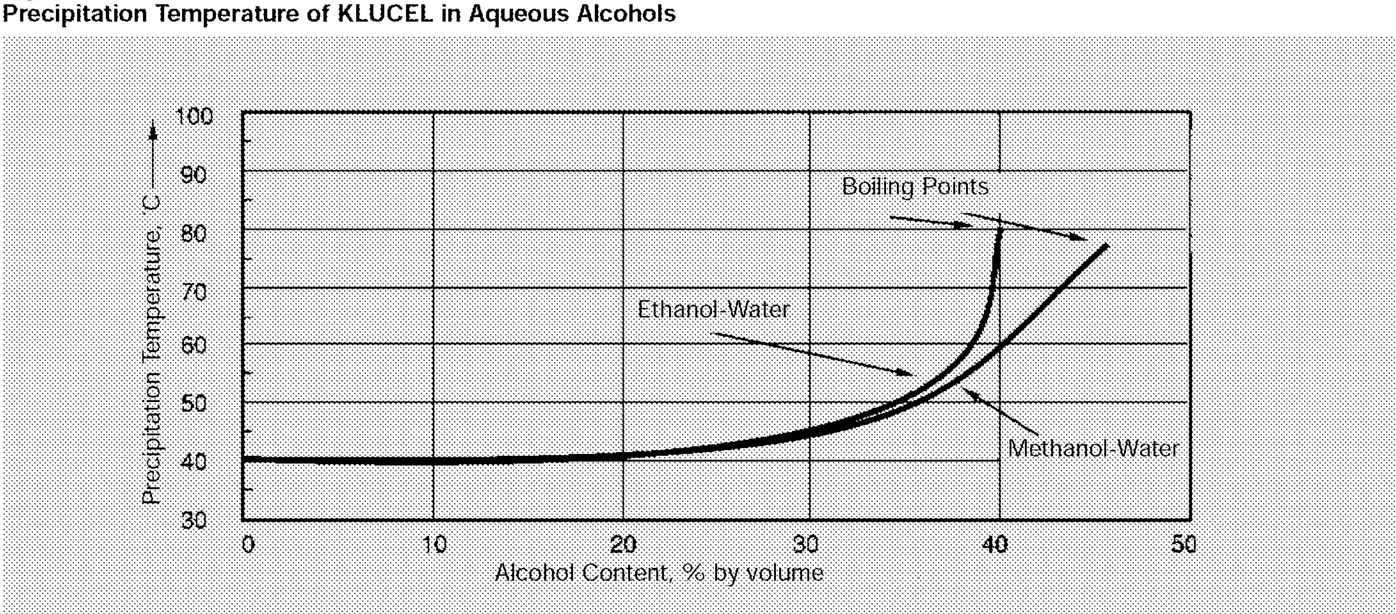



**Figure 6**
**Precipitation Temperature of KLUCEL in Aqueous Alcohols**



# THERMOPLASTICITY – MOLDING AND EXTRUSION

KLUCEL® HPC is an excellent thermoplastic polymer that can be processed by virtually all fabrication methods used for plastics. Injection- and compression-molding; blow-molding; injection foam molding; vacuum-forming; and extrusion of film, sheet, foam profiles, and filament have been demonstrated on conventional plastics equipment, using pelletized molding powder.

All molecular weight types are thermoplastic and can be readily molded and extruded. In general, the low-molecular-weight types are preferred in injection- and blow-molding, where rigidity, hardness, and dimensional stability are most important. The medium- to high-molecular-weight types are recommended for most extrusion systems where greater flexibility and higher tensile properties are desired. When fillers or extenders are used, the higher-molecular-weight types of KLUCEL are selected for their superior binding properties and their ability to maintain good flexibility and toughness, even at filler levels up to 90% by weight.

**FORMULATION OF MOLDING COMPOUNDS**

While not essential, small amounts of plasticizers are normally used with KLUCEL to ensure smooth, uniform melt flow and homogeneous end products. The addition of plasticizers will give finished articles softer and more flexible characteristics. Plasticizers such as propylene glycol, glycerin, polyethylene glycols, and trimethylolpropane are generally suitable.

Internal lubricants are added to KLUCEL molding powders to provide lubrication at the die wall or mold wall, ensuring easy release from mold surfaces and preventing lamination of the KLUCEL to itself, as in a roll of KLUCEL hydroxypropylcellulose film. Glycerol monostearate, silicones, lecithin, and various stearates have proved useful.

Antioxidants also can be added to the low-molecular-weight types to prevent discoloration and thermal degradation. Butylated hydroxytoluene, lauryl thiodipropionate, and ascorbic acid are a few of the effective antioxidants commonly used.

Fillers, extenders, and compatible polymers may be added to KLUCEL to impart some functional property, to vary physical characteristics, or to reduce costs. In general, any filler material that will withstand the melt temperatures of KLUCEL can be incorporated and processed as with other plastics. Numerous filler materials, such as starch, talc, detergents, fragrances, pigments, fertilizers, various food products, and some drugs and medicinals, have been successfully evaluated in molding and extrusion systems. Depending on the particle size and bulk density of the filler, loadings of 40 to 95 weight % are possible with KLUCEL.

A twin-screw extruder is recommended for the preparation of pelletized molding powders. A single-screw extruder can be used if it is equipped with a nylon screw or metering screw having a long, deep-flighted feed section.

Most articles fabricated from KLUCEL are custom-formulated to obtain the desired physical properties and solubility rate required for a specific application. We suggest that you contact your Aqualon sales representative for assistance in formulating a product to meet your needs.

# FILMS AND COATINGS

The excellent film properties of KLUCEL® HPC make it a useful material for fabrication of film and sheet. It is also useful in the formulation of coatings on paper, fabrics, food products, and other substrates. Flexible packaging films and sheet can be produced with conventional extrusion techniques. Coatings can be applied by extrusion or from aqueous or organic solvent systems.

**FILM PROPERTIES**
All films of KLUCEL are characterized by the following outstanding properties:

- Excellent flexibility
- Lack of tackiness at high humidity
- Good heat sealability
- Barrier to oil and fat

**PLASTICIZERS FOR FILMS**
Since films of KLUCEL® HPC are inherently flexible, it is not necessary to add plasticizers to cast films. However, in extruded films, plasticizers provide desirable die lubrication, reduction in melt viscosity, and improvement in melt uniformity. In all films of KLUCEL, added plasticizer will soften the film, reduce heat-seal temperatures, and impart improved flexibilty and increased elongation. Plasticizer levels of 5% or less (usually 2% or less) are recommended. Higher addition levels lead to reduced tensile properties, increased tackiness at high humidity, and poorer clarity, dimensional stability, and solubility characteristics.

The following plasticizers are compatible in films of KLUCEL at a concentration of 5%:
- Propylene glycol
- Glycerin
- Trimethylolpropane
- Polyethylene glycols

For improved resistance to tackiness at high humidity, the following lubricants are recommended:
- Polyethylene glycols (1,000 to 20,000 molecular weight)
- Propylene glycol monostearate
- Glycerol monostearate
- Silicone

**ADDITIVE AND MODIFIER FOR FILMS AND COATINGS**
The good film properties and wide range of solubility of KLUCEL make it a useful additive and modifier for other films and coatings. Solubility in polar organic solvents permits addition to many organic-solvent-soluble resins, while a broad range of compatibility ensures homogeneous films of improved quality.

The addition of KLUCEL in coatings generally:
- Improves flexibility.
- Improves toughness.
- Improves heat-seal properties.
- Reduces water resistance.
- Reduces the tendency to crack.

**SOLUTION CASTING FOR FILMS AND COATINGS**
The solubility of KLUCEL in water as well as in polar organic solvents affords a wide choice in the preparation of solutions used for casting film or for coating operations.

KLUCEL is insoluble in hot water, so care must be exercised when drying aqueous coatings to avoid the "blushing" that can occur at high temperatures and that might impair the film properties.

**INSOLUBILIZING FILMS AND COATINGS**
Films and coatings of KLUCEL can be made insoluble through crosslinking with resins that are reactive with the available hydroxyl groups on the KLUCEL polymer. A number of resins are capable of performing this crosslinking reaction.

Rate of cure and extent of insolubility of the final composition are generally dependent not only on the reactivity of the insolubilizing resin, but also on the temperature of the cure, pH of the system, and the amount of crosslinking resin used. High temperature, low pH, and higher proportions of resins tend to increase the rate of cure, improve water resistance, and increase stiffness.

The crosslinked composition may possess a high degree of insolubility in water, but the composition is not completely resistant and generally swells in the presence of water. This means that films and coatings have reduced wet strength and lose toughness.

# PACKAGING, REGULATORY STATUS, TOXICOLOGY, SAFETY

## PACKAGING

KLUCEL® HPC is shipped in fiber drums with a polyethylene inner liner containing 100 lbs net.

## REGULATORY STATUS

Food grade, designated with a "F," complies with the requirements of the US FDA for direct addition to food for human consumption, as specified in the Code of Federal Regulations, Title 21, Section 172.870. Food grade Klucel also conforms to the specifications for hydroxypropylcellulose set forth in the *Food Chemicals Codex*.

Personal Care grade, designated with "CS," conforms to the specifications for hydroxypropylcellulose set forth in the CTFA.

Pharmaceutical grade, designated with "F Pharm," conforms to the specifications for hydroxypropylcellulose set forth in the *U.S. National Formulary, European Pharmacopoeia* and the *Japanese Pharmacopoeia*.

## TOXICOLOGY

Toxicity testing indicates that KLUCEL is physiologically inert. The results of repeat-insult patch tests on humans disclose no evidence that it is either a primary skin irritant or skin-sensitizing agent. Feeding of KLUCEL to rats for 90 days at dietary levels up to and including 5.0% produced no gross or histopathologic changes or other deleterious effects. KLUCEL is considered nutritionally equivalent to purified cellulose in that neither material is metabolized. For a complete report, request a copy of Bulletin T-122, "KLUCEL® Hydroxypropylcellulose: Summary of Toxicological Investigations."

## PRODUCT SAFETY

KLUCEL is a flammable dust when finely divided and suspended in air. An explosion can occur if the suspended dust is ignited. Proper design and operation of facilities and good housekeeping practices can minimize this hazard.

Floors subject to spills or dusting with KLUCEL can become slippery when wetted with water. Follow good housekeeping practices and clean up spills promptly.

Read and understand the Material Safety Data Sheet (MSDS) before using this product.

| | |
|---|---|
| CASRN: | 9004-64-2 |
| CAS Name: | Cellulose 2-hydroxypropyl ether |

# APPENDIX

## METHODS OF ANALYSIS

All types of KLUCEL® HPC meet certain specifications for moisture, viscosity, and ash content. Detailed descriptions are given of Aqualon methods for determining these values.

### A. Moisture

1. Weigh duplicate samples of 5 g, to the nearest 0.001 g, into previously dried and weighed moisture cans with covers.
2. Place the samples in a gravity convection oven maintained at 105° ± 0.5°C. Heat for 3 hrs. Cool in a desiccator and weigh.
3. Return the sample to the oven for 45 min. Cool and weigh as before. If the second dried weight is not within 0.005 g of the first dried weight, repeat the 45-min oven periods until two subsequent weighings are in agreement. Then, using the lowest dried weight obtained, calculate percent moisture as follows:

$$\frac{\text{Original sample weight} - \text{dry sample weight}}{\text{Original sample weight}} \times 100 = \%\ \text{moisture}$$

### B. Viscosity in Water

As explained on page 9, the apparent viscosity of a solution of KLUCEL depends on a number of factors. If reproducible results are to be obtained, a closely standardized method of solution preparation and viscosity determination must be followed.

Preparation of the solution is critical in that KLUCEL must be completely dissolved in order to obtain a significant measurement. In weighing out the proper amount of KLUCEL for a viscosity determination, care must be taken to include a moisture correction. This correction compensates for the moisture in KLUCEL and places the viscosity measurement of KLUCEL on a dry basis. The viscosity-measurement test must be rigidly standardized because the viscosity reading obtained is dependent on the rate of shear during dissolution, the amount of agitation prior to measurement, the temperature, and the elapsed time between agitation and measurement. The method used in Aqualon laboratories is therefore given here in detail.

***Solution Preparation***

Immediately after taking portions of the KLUCEL polymer sample for moisture determination, portions of the same undried KLUCEL should be taken for viscosity solution preparation. Weighings of moisture and solution samples should be carried out practically together to ensure that the moisture content of the respective portions is the same at time of weighings.

The standardized Aqualon method for determining the viscosity of solutions of KLUCEL specifies use of the Brookfield viscometer, Model LVF (Brookfield Engineering Labs, Stoughton, Massachusetts; 4 spindles, 4 speeds covering the range 0 to 100,000 cps).

The solution volumes specified under item 4 should not be less than outlined or there may not be sufficient solution to cover the appropriate Brookfield spindle.

1. Weigh the specified amounts to ± 0.005 g, as obtained from Table XIII, page 24, quickly into clean, ground-glass-stoppered weighing bottles. Stopper immediately to eliminate gain or loss of moisture content.
2. From the determined percent moisture, calculate the water to be added for the respective viscosity solutions, as follows:

   a. For a 1% viscosity solution:

   $$(\text{Weight of KLUCEL, g}) \times (99 - \%\ \text{moisture}) = \text{Weight of water, g}$$

   b. For a 2% viscosity solution:

   $$\frac{(\text{Weight of KLUCEL, g}) \times (98 - \%\ \text{moisture})}{2} = \text{Weight of water, g}$$

   c. For a 5% viscosity solution:

   $$\frac{(\text{Weight of KLUCEL, g}) \times (95 - \%\ \text{moisture})}{5} = \text{Weight of water, g}$$

   d. For a 10% viscosity solution:

   $$\frac{(\text{Weight of KLUCEL, g}) \times (90 - \%\ \text{moisture})}{10} = \text{Weight of water, g}$$

3. Add the calculated amount of distilled water to a 16-oz bottle.
4. Stir the water with a mechanical agitator to create a vortex, and slowly sift the KLUCEL into the vortex over a 15 to 30-sec period to ensure good dispersion. An anchor-shaped stirrer turned by a compressed-air or electric motor has been found to be satisfactory. After the solution appears to be complete, stir it for an additional 10 to 15 min at high speed. Be careful to avoid loss of solution.
5. When the solution is complete, cover the mouth of the bottle with cellophane and screw the cap on securely. Place it in a constant-temperature bath (25° ± 0.2°C) for 30 min, or for as long as necessary to adjust the solution temperature to 25° ± 0.2°C.

***Viscosity Determination***

6. While the solution is in the constant-temperature bath, select from Table XIII below the Brookfield spindle-speed combination corresponding to the viscosity type of KLUCEL® HPC being tested. Determine the viscosity of the KLUCEL within 2 hrs after removing it from the stirrer. If the solution stands longer than 2 hrs, return it to the stirrer for 10 min, place it in a bath for 30 min, and then determine viscosity.
7. Remove the bottle from the constant-temperature bath and stir the solution by hand for 10 sec, using a stirring rod. Avoid shaking or vigorous stirring, as this will increase air bubbles, which interfere with viscosity measurement.
8. Immediately insert the appropriate Brookfield viscometer spindle into the solution and start the spindle rotating. Allow it to rotate for 3 min before taking the reading.
9. Stop the instrument and read the dial. Multiply the dial reading by the factor corresponding to the speed and spindle used. The result is the viscosity of the solution in centipoises.

**C. Viscosity in Ethanol**

The viscosity of ethanol solutions of KLUCEL is determined in the same manner as for aqueous solutions, but with the following modification:

1. To minimize evaporation of the ethanol, the bottle may be capped and mechanically shaken to accomplish solution of the KLUCEL instead of stirring the solution by hand.
2. Repeat steps 5, 6, 7, 8, and 9 as described in the Viscosity in Water procedures.

**D. Ash Content**

The ash components of KLUCEL are sodium salts. Determination of these is complicated by the presence of certain anticaking agents, which contribute to the total ash. The ash method is referenced in the *National Formulary*, current edition.

Table XIII – Sample Weights of KLUCEL, and Brookfield Spindle and Speed for Viscosity Measurement

| Viscosity Type | Concentration, % rpm | Sample Wt, g | Brookfield Settings | |
|---|---|---|---|---|
| | | | Spindle No. | |
| E 10 | 25.0 | 30 | 2 | |
| L 5 | 11.0 | 30 | 1 | |
| J 5 | 11.0 | 60 | 2 | |
| G 2 | 5.2 | 60 | 2 | |
| M 2 | 5.2 | 60 | 4 | |
| H 1 | 2.3 | 30 | 3 | |

# PRODUCT LISTING SUPPLEMENT

The following list of products, along with their chemical identity and source of supply, may be helpful to the reader who is unfamiliar with some of the products referred to in this booklet.

Read and understand the Material Safety Data Sheets (MSDSs) before using these products.

| Product | Chemical Identity | Manufacturer |
|---|---|---|
| Antifoam AF | Dimethylpolysiloxane materials | Stewart Hall Chemical |
| Aqualon® CMC | Sodium carboxymethylcellulose | Hercules Incorporated |
| Benecel® MC | Methylcellulose | Hercules Incorporated |
| Butyl Cellosolve | Ethylene glycol monobutyl ether-solvent | Dow Chemical Co. |
| Carbowax 1000 | Polyethylene glycol | Dow Chemical Co. |
| Cellosolve | Ethylene glycol monoethyl ether-solvent | Dow Chemical Co. |
| Colloid 581-B | Hydrocarbon-based material containing surfactants, waxes, and metallic soaps | Rhône-Poulenc, Inc. |
| Defoamer 1512 | Silica-type defoamer | Hercules Incorporated |
| Methyl Cellosolve | Ethylene glycol monomethyl ether | Dow Chemical Co. |
| M-Pyrol | N-methyl-2-pyrrolidone | GAF Corporation |
| Myvacet | Acetylated monoglyceride | Eastman Chemical Products, Inc. |
| Myverol | Monoglyceride | Eastman Chemical Products, Inc. |
| Natrosol® HEC | Hydroxyethylcellulose | Hercules Incorporated |
| Nopco KFS | Proprietary blend of hydrocarbon oil, fatty acids, wax, and nonionic surfactants | Henkel Corporation |
| Nopco NDW | Proprietary blend of hydrocarbon oils, silicone, nonionic surfactants, and metallic soaps | Henkel Corporation |
| Omadine | Sodium 1-hydroxpyridine-2-thione | Olin Chemicals |

250-2F REV. 10-01 500 Supersedes all previous editions. PRINTED IN U.S.A.